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Polynitrogen Chemistry. Synthesis, Characterization, and Crystal Structure of Surprisingly Stable Fluoroantimonate Salts of N_5^+

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Uneven margin (or were you centering this?)

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Abstract

The new N_5^+ salt, $N_5^+SbF_6^-$, was prepared from $N_2F^+SbF_6^-$ and HN_3 in anhydrous HF solution. The white solid is surprisingly stable, decomposing only at 70 °C, and is relatively insensitive to impact. Its vibrational spectrum exhibits all nine fundamentals with frequencies that are in excellent agreement with the theoretical calculations for a five-atomic V-shaped ion of C_{2v} symmetry. The $N_5^+Sb_2F_{11}^-$ salt was also prepared and its crystal structure was determined. The geometry previously predicted for free gaseous N_5^+ from theoretical calculations was confirmed within experimental error. The $Sb_2F_{11}^-$ anions exhibit an unusual geometry with eclipsed SbF_4 groups due to inter-ionic bridging with the N_5^+ cations. The N_5^+ cation is a powerful one-electron oxidizer. Its electron affinity falls between 11.0 and 12.08 eV because it readily oxidizes NO to NO^+ and NO_2 to NO_2^+ but fails to oxidize Xe or O_2.

Introduction

The recent discovery of $N_5^+AsF_6^-$ as a marginally stable compound that can be prepared on a macroscopic scale is quite remarkable.[1] It represented only the third readily accessible homoleptic polynitrogen compound besides N_2 and N_3^- and as such has received much public acclaim.[2] Since N_5AsF_6 is only marginally stable and had given rise to some explosions,[1] it was of great interest to search for more stable N_5^+ salts in order to allow a more thorough characterization of this fascinating cation and to provide a suitable starting material for the pursuit of nitrogen allotropes. In this paper, the synthesis and characterization of surprisingly stable fluoroantimonate salts of N_5^+ and the crystal structure of $N_5^+Sb_2F_{11}^-$ are reported.

Experimental

Caution! *HN_3, azides and polynitrogen compounds are highly endothermic and can decompose explosively. They should be handled only on a small scale with appropriate safety precautions (face shields, leather gloves, and protective clothing). Condensation of neat HN_3 at – 196 °C into Teflon ampoules containing oxidizers has repeatedly resulted in explosions upon condensation or melting of the HN_3.*

Materials and Apparatus. All reactions were carried out in ¾ inch o. d. Teflon-FEP or -PFA ampoules that contained Teflon coated magnetic stirring bars and were closed by stainless steel valves. Volatile materials were handled on a stainless steel / Teflon-FEP vacuum line.[3] Nonvolatile solids were handled in the dry nitrogen atmosphere of a glove box. HN_3 was generated and handled on a Pyrex glass vacuum line

equipped with grease-free Kontes glass-Teflon valves. Infrared spectra were recorded on a Mattson Galaxy FT-IR spectrometer using dry powders pressed between AgCl windows in an Econo press (Barnes Engineering Co.). Raman spectra were recorded on a Bruker Equinox 55 FT-RA spectrometer using a Nd-Yag laser at 1064 nm and Pyrex melting point capillaries as sample containers. The thermal stabilities were determined using a DuPont Model 910 DSC, crimp-sealed aluminum pans as sample containers, and heating rates of 3 °C/min. The data were recorded and analyzed with a DuPont Model 2000 thermal analyst. Impact sensitivities were measured on an Olin Mathieson drop weight tester standardized with RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine, 30 kg·cm, 50 %).

The $N_2F^+SbF_6^-$ and $N_2F^+Sb_2F_{11}^-$ starting materials were prepared from *cis*-N_2F_2 and SbF_5 in anhydrous HF solution as previously described.[4-7] The HF (Matheson Co.) was dried by storage over BiF_5 (Ozark Mahoning).[8] The NO and NO_2 (Matheson Co) were purified by fractional condensation prior to their use. The O_2 and Xe (Matheson Co) were used as received. The preparation of HN_3 has previously been described.[1]

Preparation of $N_5^+SbF_6^-$. A Teflon ampoule, equipped with a stainless steel valve and containing a Teflon-coated magnetic stirring bar, was passivated with ClF_3. It was attached to the metal vacuum line and treated several times with anhydrous HF until no color was observed upon freezing the HF at –196 °C. It was then loaded with $N_2F^+SbF_6^-$ (4.97 mmol) in the glove box and attached to the metal vacuum line. The ampoule was evacuated and cooled to –196 °C. Anhydrous HF (~2 mL) was then condensed into the ampoule and its contents were allowed to warm to ambient temperature with occasional stirring. After all the $N_2F^+SbF_6^-$ had dissolved, the ampoule

was re-cooled to –196 °C and some additional neat HF was condensed onto the upper walls of the tube where the HN_3 was going to be frozen out. The cold ampoule was then connected to the glass line, and HN_3 (5.00 mmol) was added slowly at –196 °C. The reaction mixture was allowed to warm slowly behind a safety shield to room temperature and kept at this temperature for about 45 min. The volatile materials were removed by pumping for several hours at 20 °C, leaving behind a white powder (1.502 g, weight calcd for 4.97 mmol of N_5SbF_6 = 1.520 g) that was identified by its vibrational spectra as N_5SbF_6.

This reaction was also carried out by first condensing HN_3 at –196 °C into a prepassivated and preweighed Teflon ampoule containing a known amount of HF. The resulting mixture was homogenized at ambient temperature. The ampoule was taken into the glove box, where a stoichiometric amount of $N_2F^+SbF_6^-$ was added at –196 °C. The cold ampoule was attached to the metal vacuum line and evacuated. Subsequent slow warming of the reaction mixture to room temperature for about 30 min, followed by removal of all volatile material resulted in the isolation of $N_5^+SbF_6^-$ in >99% yield.

Preparation of $N_5^+Sb_2F_{11}^-$. Freshly distilled SbF_5 (1.449 mmol) was added in the glove box to a prepassivated Teflon-FEP ampoule, and HF (1.9 mL liquid) was added on the metal vacuum line at –196 °C. The mixture was homogenized at room temperature and taken back into the glove box. The ampoule was cooled inside the glove box to –196 °C and opened, and $N_5^+SbF_6^-$ (1.444 mmol) was added. The resulting mixture was allowed to warm to room temperature, and all volatile material was pumped off. The white solid residue (758 mg, weight calcd. for 1.444 mmol of $N_5^+Sb_2F_{11}^-$ = 755 mg) was shown by vibrational spectroscopy to consist of $N_5^+Sb_2F_{11}^-$.

Reactions of $N_5^+SbF_6^-$ with NO, NO_2, O_2 or Xe. In a typical experiment, a .5 inch Teflon-FEP ampoule, that was closed by a Teflon valve, was loaded in the dry-box with $N_5^+SbF_6^-$ (.53 mmol) . On the vacuum line, NO (4.2 mmol) was added at –196 °C and the contents of the ampoule were allowed to warm slowly with intermittent cooling to room temperature. After keeping the ampoule for 2 hr at room temperature, it was cooled back to –196 °C and the volatile gas (1.34 mmol of N_2) was measured and pumped off. The amount of unreacted NO was measured (3.6 mmol) and pumped off at room temperature, leaving behind .53 mmol of $NO^+SbF_6^-$ that was identified by vibrational spectroscopy.

In a similar manner, $N_5^+SbF_6^-$ was found to react quantitatively with NO_2, but no reaction was observed with either Xe or O_2.

Crystal structure determination of $N_5^+Sb_2F_{11}^-$. About 1 mL of anhydrous SO_2 was condensed onto 0.200 g of N_5SbF_6 at –196 °C in a ½ inch o. d. sapphire tube (Tyco Corp.) closed by a stainless steel valve. The contents of the tube were warmed to –78 °C causing all of the N_5SbF_6 to dissolve and form a pale yellowish solution. Anhydrous SO_2ClF (~1.5 mL) was then slowly condensed onto this solution under vacuum. The solvents were then slowly removed under a static vacuum at –64 °C over a period of ~16 hours leaving behind plate-like colorless crystals. These crystals were extremely reactive to perfluoropolyether oil and showed an instantaneous evolution of nitrogen gas. The majority of the crystals were very soft and difficult to handle, but a few crystals appeared to exhibit a different habit and better mechanical strength. One of these crystals was immersed in halocarbon grease and mounted on the goniometer head using a precentered Nylon Cryoloop equipped with a magnetic base. The structure of the salt was determined

using a Bruker diffractometer equipped with a CCD detector and a low temperature, LT3, device. The 3-circle platform with a fixed χ-axis was controlled by the SMART[9] software package. The unit cell parameters were determined at –60 °C from three runs of data with 30 frames per run using a scan speed of 30-seconds per frame. A complete hemisphere of data was collected, using 1271 frames at 30 sec/frame, including 50 frames that were collected at the beginning and end of the data collection to monitor crystal decay. Data were integrated using the SAINT[10] software package, and the raw data was corrected for absorption using the SADABS[11] program. The absence of $h + k$ = odd and $h0l$ reflections (l = odd) shows the presence of a C-centered lattice and a c-glide plane parallel and perpendicular to the b-axis, respectively, indicating Cc or C2/c as the likely space groups. The intensity statistics, E^2-1 values, indicated a centrosymmetric space group thereby excluding *Cc* as a possible space group. The space group was thus unambiguously assigned as *C2/c*. The structure was solved by the Patterson method using the SHELXS-97[12] program and refined by the least squares method on F^2 using SHELXL-97.[13] The initial Patterson map revealed the position of the two Sb atoms linked by a fluorine atom. The remaining atoms were located from subsequent difference electron density maps and finally refined anisotropically by the least-squares method on F^2 using the SHELXTL[14] 5.1 software for Windows NT. The crystal did not show any significant decomposition during the data collection. The experimental and refinement parameters, and the atomic coordinates and thermal displacement parameters are listed in Tables 1 and 2, respectively.

Results and Discussion

Synthesis and Properties of $N_5^+SbF_6^-$. The synthesis of $N_5^+SbF_6^-$ was carried out in the same manner as previously reported[1] for $N_5^+AsF_6^-$ by reacting $N_2F^+SbF_6^-$ with HN_3 in anhydrous HF solution at –78 °C, followed by removal of the volatile products at room temperature.

$$N_2F^+SbF_6^- \quad + \quad HN_3 \longrightarrow N_5^+SbF_6^- \quad + \quad HF$$

The yield of $N_5^+SbF_6^-$ is essentially quantitative and the product purity is high. It is essential that the reaction system is completely anhydrous as water hydrolyzes the N_5^+ salt generating free SbF_5 which in combination with HF protonates HN_3 under formation of $H_2N_3^+SbF_6^-$.[15]

The $N_5^+SbF_6^-$ salt is a colorless hygroscopic solid that is stable at ambient temperature and, based on the DSC data, starts to decompose at 70 °C. It is surprisingly insensitive to impact. Even at the maximum setting of our apparatus (200 kg·cm), only partial thermal decomposition due to adiabatic heating of the sample but no explosions were observed. The salt is soluble in and compatible with HF, SO_2, and CHF_3.

The oxidizing properties of $N_5^+SbF_6^-$ were examined in the solid state and in HF solution by exposing it to 2 atmospheres of either oxygen or xenon gas between –78 °C and ambient temperature. No oxidations to O_2^+ and Xe_2^+, respectively, were observed under these conditions, showing that the electron affinity of N_5^+ is lower than the first ionization potential of xenon (12.08 eV), *i. e.*, N_5^+ is a weaker oxidizer than either PtF_6 that can oxidize O_2 to O_2^+ [16] or O_2^+ that can oxidize Xe to Xe_2^+ under similar conditions.[17,18] In spite of the first ionization potential of N_2 (15.51 eV) being 3.01 eV higher than that of O_2 (12.5 eV), the electron affinity of N_5^+ is lower than that of O_2^+

because in N_5^+ the positive charge is spread over a larger number of atoms, thereby decreasing its oxidizing power. However, it was found that N_5^+ can quantitatively oxidize either NO (1. IP = 9.5 eV) or NO_2 (1. IP = 11.0 eV) according to:

$$NO + N_5^+SbF_6^- \longrightarrow NO^+SbF_6^- + 2.5\ N_2$$

and

$$NO_2 + N_5^+SbF_6^- \longrightarrow NO_2^+SbF_6^- + 2.5\ N_2$$

Therefore the electron affinity of N_5^+ must have a value between 11.0 and 12.08 eV, rendering it a very strong one-electron oxidizer. Although it is not quite as powerful as PtF_6 or O_2^+ salts, it offers the great advantage of not acting as a fluorinating or oxygenating agent, which can be a very important consideration when dealing with substrates that are easily fluorinated or oxygenated.

Ongoing studies in our laboratory show that the potential hazards of handling neat HN_3 in the synthesis of N_5^+ can be avoided by either replacing HN_3 with the insensitive $(CH_3)_3SiN_3$ or generating the desired HN_3 from a weighed amount NaN_3 and excess HF in a separate ampoule and transferring all volatiles into the reaction vessel containing an HF solution of $N_2F^+SbF_6^-$. The reactions with $(CH_3)_3SiN_3$ are carried out in either HF or SO_2 solution and produce N_5^+ in high yield. When HF is used as the solvent, the first reaction step most certainly involves the formation of $(CH_3)_3SiF$ and HN_3, *i. e.*, HN_3 is generated *in situ* in the reactor.

Synthesis and Properties of $N_5^+Sb_2F_{11}^-$. To preclude a potential side reaction of $Sb_2F_{11}^-$ with HF and HN_3 to give SbF_6^- and $H_2N_3^+SbF_6^-$, a sample of $N_5^+SbF_6^-$ was reacted with an equimolar amount of SbF_5 in HF solution at room temperature.

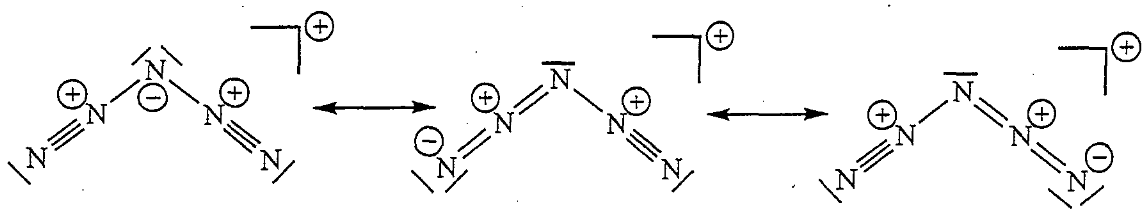
$$N_5^+SbF_6^- \quad + \quad SbF_5 \longrightarrow N_5^+Sb_2F_{11}^-$$

The resulting $N_5^+Sb_2F_{11}^-$ salt is a colorless solid that is stable at room temperature and undergoes, according to its DSC data, thermal decomposition at 70 °C, *i. e.*, its thermal stability is comparable to that of $N_5^+SbF_6^-$ but, contrary to $N_5^+SbF_6^-$, it undergoes a reversible endotherm (melting) at about 30 °C. Consequently, the replacement of SbF_6^- by $Sb_2F_{11}^-$ did not result in increased thermal stability and does not appear to offer any significant advantages for studying the reaction chemistry of N_5^+ salts.

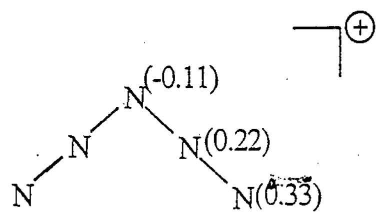
Crystal Structure of $N_5^+Sb_2F_{11}^-$. The structure of $N_5^+Sb_2F_{11}^-$ is shown in Fig. 1-4 and the important bond lengths and angles are summarized in Table 3. The observed V-shaped geometry of the N_5^+ cation is in excellent agreement with the theoretical predictions[1] for the free gaseous N_5^+ cation at the B3LYP level of theory with the calculated terminal and central N-N bond distances of 1.11 and 1.30 Å being close to the observed ones of 1.105(19) and 1.299(19) Å, respectively. Furthermore, the terminal N-N distance of 1.105(19) Å in N_5^+ is only slightly longer than that of 1.089(9) Å found for N_2F^+ in $N_2F^+Sb_2F_{11}^-$,[19] and compares well with the N-N bond distances of 1.0976(2) Å in N_2[20] and 1.0927 Å found in HN_2^+,[21,22] indicating that these bonds approximate triple bonds. The central N-N bond length of 1.299(19) Å in N_5^+ is somewhat longer than those found for typical N-N double bonds (1.17 to 1.25 Å), but is significantly shorter than those found for typical N-N single bonds (1.43 to 1.75 Å).[23] Also, the agreement between

calculated [112.3 and 166.7°] and observed [111.2(11) and 167.2(15)°, respectively] bond angles is very good.

The observed geometry supports the previously given rationale[1] that the exceptional stability of N_5^+ is largely due to resonance stabilization, resulting in relatively high bond orders for all the bonds. The bonding in N_5^+ can be described by the following three resonance structures:



Although the relative contributions from (I) and the equivalent pair, (II) and (III), are unknown, the calculated charge distributions at the NBO(B3LYP/aug-cc-pVDZ) level[24]



and the relative shielding of the N-nmr signals (the shielding increases from the terminal nitrogen to the β-nitrogen to the central nitrogen in accord with the calculated charge distributions)[1] strongly indicate that the terminal and the β-nitrogen atoms carry the positive charges and the central nitrogen a small negative one.

A least-squares-plane analysis for N_5^+ shows that the cation is essentially planar. The N2 atom exhibits a maximum deviation of 0.11 Å from the average mean plane that shows a root mean square deviation of 0.0058 Å. The N_5^+ mean plane is almost

perpendicular (78.1°) to the plane containing the F5-Sb1-F6-Sb2-F11 atoms. The latter is also almost perfectly planar and shows a root mean square deviation of 0.014 Å.

The geometry of the $Sb_2F_{11}^-$ anion also deserves a special comment. This anion is known to possess little rigidity and can exist in either an eclipsed or staggered conformation and exhibit a wide range of Sb-F-Sb bridge angles, depending upon the counter ion present in the crystal lattice.[25] The eclipsed conformation is rare but has previously also been observed for $BrF_4^+Sb_2F_{11}^-$.[19] In the latter compound, the eclipsed structure results from a packing effect in which one equatorial fluorine ligand of each antimony atom of $Sb_2F_{11}^-$ bridges to a different BrF_4^+ cation. Since the two BrF_4^+ cations and the $Sb_2F_{11}^-$ anion are coplanar, the bridging equatorial fluorine ligands around the antimonies become also coplanar resulting in the eclipsed configuration. The eclipsed conformation of the $Sb_2F_{11}^-$ anion found for $N_5^+Sb_2F_{11}^-$ (Fig. 2) is also due to fluorine bridging but results from N_5^+ acting as a spacer between the two equatorial SbF_4 units of $Sb_2F_{11}^-$ (Fig. 3). In accord with the resonance structures and the calculated charge distributions of N_5^+ (see above), the positively charged β-nitrogen atoms interact with the negatively charged fluorine ligands forming bridges that are shorter than the sum of the van der Waals radii of 3.0 Å (Figures 2-4). Thus, N2 and N4 straddle the two eclipsed fluorine atoms, F2 and F8, but since the N_5^+ plane is not perfectly perpendicular to the F_{ax}-Sb-F-Sb-F_{ax} plane, the N2-F8 = 2.723(15) Å and N4-F2 = 2.887(16) Å distances are somewhat shorter than those of N2-F2 = 3.032(16) Å and N4-F8 = 2.993(16) Å. In addition, N2 and N4 bridge to two fluorine atoms from other $Sb_2F_{11}^-$ anions with N4-F4B = 2.813(15) Å and N2-F10A = 2.768(15) Å. In contrast to $BrF_4^+Sb_2F_{11}^-$, which has an

almost linear Sb-F-Sb bridge angle of 175°,[19] that of 155.0(4)° in $N_5^+Sb_2F_{11}^-$ is much closer to those usually found for $Sb_2F_{11}^-$.[25]

Vibrational Spectra of N_5^+. The infrared and Raman spectra of solid $N_5^+SbF_6^-$ are shown in Figures 5 and 6, respectively. The experimentally observed frequencies of $N_5^+SbF_6^-$, $N_5^+Sb_2F_{11}^-$, and $N_5^+AsF_6^-$, together with their assignments, are listed in Table 4. A comparison of the observed and calculated frequencies and intensities of N_5^+ is given in Table 5. As can be seen, the previously missing[1] remaining 4 fundamental vibrations and numerous combination bands of N_5^+ have been observed and are in excellent agreement with the theoretical predictions for point group C_{2v}. The splittings observed for $\nu_8(B_2)$ and $\nu_2(A_1)$ can be accounted for by Fermi resonance. The presence of $Sb_2F_{11}^-$ impurities in the SbF_6^- salt can be readily detected by Raman bands at 692, 598, and 231 cm^{-1} and infrared bands at 708 and 497 cm^{-1} that are characteristic for $Sb_2F_{11}^-$ and do not overlap with the SbF_6^- bands.

Conclusion

The synthesis and thorough characterization of $N_5^+SbF_6^-$ and $N_5^+Sb_2F_{11}^-$ demonstrate that the N_5^+ cation can form exceptionally stable salts with fluoroantimonate anions and that these salts are surprisingly insensitive to impact. The N_5^+ cation is a powerful one-electron oxidizer with an electron affinity between 11.0 and 12.08 eV while not giving rise to undesirable fluorination or oxygenation side-reactions. The ready availability of a stable polynitrogen cation in addition to the long known azide anion opens a venue to neutral polynitrogen compounds and may provide the basis for the first synthesis of nitrogen allotropes.

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Supporting Information Available

Tables of structure determination summary, atomic coordinates, bond lengths and angles and anisotropic displacement parameters of $N_5Sb_2F_{11}$ in CIF format. This material is available free of charge via the internet at http://pubs.acs.org.

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Table 1. Crystal data and structure refinement for $N_5^+Sb_2F_{11}^-$

Identification code	$N_5^+Sb_2F_{11}^-$
Empirical formula	F11 N5 Sb2
Formula weight	522.55
Temperature	213(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions	a = 10.913(8) Å α = 90°.
	b = 12.654(8) Å β = 104.715(18)°.
	c = 16.675(11) Å γ = 90°.
Volume	2227(3) $Å^3$
Z	8
Density (calculated)	3.117 Mg/m^3
Absorption coefficient	4.995 mm^{-1}
F(000)	1888
Crystal size	0.26 x 0.10 x 0.05 mm^3
Theta range for data collection	2.51 to 25.35°.
Index ranges	-12<=h<=13, -15<=k<=15, -20<=l<=17
Reflections collected	9125
Independent reflections	2022 [R(int) = 0.0629]
Absorption correction	Sadabs
Max. and min. transmission	0.7883 and 0.3567
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2022 / 0 / 164
Goodness-of-fit on F^2	1.122
Final R indices [I>2sigma(I)]	R1 = 0.0678, wR2 = 0.1913
R indices (all data)	R1 = 0.0785, wR2 = 0.2019
Extinction coefficient	0.00026(18)
Largest diff. peak and hole	4.329 and -2.102 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $N_5^+Sb_2F_{11}^-$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Sb(1)	2327(1)	5268(1)	5963(1)	32(1)
Sb(2)	1229(1)	2343(1)	6199(1)	28(1)
F(1)	930(11)	5546(9)	6373(7)	65(3)
F(2)	3187(10)	4839(8)	7017(5)	59(3)
F(3)	3652(9)	4765(7)	5561(6)	48(2)
F(4)	1368(9)	5453(7)	4889(5)	51(2)
F(5)	2906(11)	6638(7)	6101(6)	64(3)
F(6)	1691(8)	3755(6)	5813(5)	43(2)
F(7)	-17(9)	3062(8)	6548(6)	53(2)
F(8)	2340(9)	2672(7)	7217(5)	50(2)
F(9)	2592(10)	1837(9)	5844(6)	63(3)
F(10)	190(10)	2218(6)	5142(5)	49(2)
F(11)	854(10)	1003(6)	6539(6)	59(3)
N(1)	4750(14)	3132(16)	8389(9)	62(4)
N(2)	3965(12)	3518(11)	8581(7)	40(3)
N(3)	3138(14)	3881(10)	8954(8)	51(4)
N(4)	2229(13)	4376(10)	8441(7)	40(3)
N(5)	1369(14)	4828(13)	8136(8)	52(4)

Table 3. Bond lengths and angles for $N_5^+Sb_2F_{11}^-$

Bond lengths (Å)

Sb(1)-F(5)	1.839(8)	Sb(2)-F(9)	1.849(10)
Sb(1)-F(4)	1.845(8)	Sb(2)-F(8)	1.866(8)
Sb(1)-F(2)	1.851(9)	Sb(2)-F(11)	1.866(8)
Sb(1)-F(3)	1.854(9)	Sb(2)-F(6)	2.007(7)
Sb(1)-F(1)	1.856(10)	N(1)-N(2)	1.102(19)
Sb(1)-F(6)	2.031(7)	N(2)-N(3)	1.303(19)
Sb(2)-F(10)	1.844(7)	N(3)-N(4)	1.295(19)
Sb(2)-F(7)	1.849(9)	N(4)-N(5)	1.107(19)

Bond Angles (°)

F(5)-Sb(1)-F(4)	95.4(4)	F(9)-Sb(2)-F(8)	88.8(5)
F(5)-Sb(1)-F(2)	94.8(4)	F(10)-Sb(2)-F(11)	94.8(4)
F(4)-Sb(1)-F(2)	169.9(4)	F(7)-Sb(2)-F(11)	96.7(5)
F(5)-Sb(1)-F(3)	95.4(5)	F(9)-Sb(2)-F(11)	92.2(5)
F(4)-Sb(1)-F(3)	89.7(4)	F(8)-Sb(2)-F(11)	93.9(4)
F(2)-Sb(1)-F(3)	89.3(5)	F(10)-Sb(2)-F(6)	85.0(3)
F(5)-Sb(1)-F(1)	93.6(5)	F(7)-Sb(2)-F(6)	85.9(4)
F(4)-Sb(1)-F(1)	91.0(5)	F(9)-Sb(2)-F(6)	85.3(5)
F(2)-Sb(1)-F(1)	88.4(5)	F(8)-Sb(2)-F(6)	86.3(4)
F(3)-Sb(1)-F(1)	170.8(4)	F(11)-Sb(2)-F(6)	177.5(4)
F(5)-Sb(1)-F(6)	179.9(5)	Sb(2)-F(6)-Sb(1)	155.0(4)
F(4)-Sb(1)-F(6)	84.7(4)	N(1)-N(2)-N(3)	168.1(15)
F(2)-Sb(1)-F(6)	85.1(4)	N(4)-N(3)-N(2)	111.2(11)
F(3)-Sb(1)-F(6)	84.5(4)	N(5)-N(4)-N(3)	166.3(14)
F(1)-Sb(1)-F(6)	86.4(4)		
F(10)-Sb(2)-F(7)	91.2(5)		
F(10)-Sb(2)-F(9)	90.8(5)		
F(7)-Sb(2)-F(9)	170.7(5)		
F(10)-Sb(2)-F(8)	171.3(4)		
F(7)-Sb(2)-F(8)	87.9(4)		

Table 4. Observed Infrared and Raman Spectra of $N_5^+SbF_6^-$, $N_5^+Sb_2F_{11}^-$ and $N_5^+AsF_6^-$ and Their Assignments

obsd freq, cm^{-1} (rel intens)						assgnts (point group)		
$N_5^+SbF_6^-$		$N_5^+Sb_2F_{11}^-$		$N_5^+AsF_6^-$				
IR	RA	IR	RA	IR	RA	$N_5^+(C_{2v})$	$MF_6^-(O_h)$	$Sb_2F_{11}^-$
3357 vw						$(\nu_1 + \nu_3 + \nu_9)(B_2) = 3358$		
3334 vw						$(\nu_1 + \nu_8)(B_2) = 3323$		
3079 w		3069 w				$(\nu_2 + \nu_7)(B_2) = 3077$		
2681 vw		2671 vw				$(\nu_1 + \nu_9)(B_2) = 2682$		
2270 m	2268 (9.4)	2260 m	2261 (9.0)	2270 m	2271 (4.4)	$(\nu_1)(A_1)$		
2205 s	2205 (2.0)	2203 s	2202 (1.9)	2210 s	2211 (0.8)	$(\nu_7)(B_2)$		
1921 vw		1919 vw				$(\nu_3 + 3\nu_9)(B_2) = 1914$		
1891 vw		1883 vw				$(\nu_8 + 2\nu_9)(B_2) = 1883$		
1240 vw		1366 w 1288 vw					Comb. bands	Comb. bands
1092 ms		1089 s		1088 s		$(\nu_3 + \nu_9)(B_2) = 1086^a$		
1064 s		1064 s				$\nu_8(B_2)$		
902 vvw		892 vvw				$(\nu_5 + \nu_6)(B_2) = 903$		
871 w	872 (0.6)	867 w	866 (0.6)	872 w	871 (0.7)	$\nu_2(A_1)$		
835 vw	837 (0+)	824 vw	824 (0+)			$(2\nu_9)(A_1) = 828^b$		
		725 – 650 vs,br	692 (5.5) 654 (10) 598 (1.4)					νSbF
	672 (1)		664 (~1)	680 sh	669/672 (1.8)	$\nu_3(A_1)$		
655 vs				704 vs			$\nu_3(F_{1u})$	
	652 (10)				686 (10)		$\nu_1(A_{1g})$	
		596 mw 537 mw						νSbF
582 w	571 (0.8)			575 w	579 (1.6)		$\nu_2(E_g)$	
		497 s						νSb-F-Sb
	478 (0+)		470 (0+)			$\nu_5(A_2)$		
447 w		449 w				?		
425 ms		417 ms		420 sh		$\nu_6(B_1)$		
412 mw	416 (0+)	409 sh	417 (0+)			$\nu_9(B_2)$		
			295 (2.1) 283 sh 272 sh 231 (2.0)					δSb-F
284 vs				394 vs			$\nu_4(F_{1u})$	
	282 (2.8)				372 (3.4)		$\nu_5(F_{2g})$	
	204 (5.0)		200 (3.6)		209 (4.4)	$\nu_4(A_1)$		
	107 (5)		135 sh 97 (5.0)		125 (5.5)	lattice vibrations		

a) In Fermi resonance with $\nu_8(B_2)$. b) In Fermi resonance with $\nu_2(A_1)$

Table 5. Comparison of Observed and Unscaled Calculated CCSD(T)/6311+G(2d) Vibrational Frequencies (cm^{-1}) and Intensities (km mol^{-1} and $Å^4 amu^{-1}$) for N_5^+

	approx mode description in point group C_{2v}	calcd freq (abs IR, Ra int)			obsd freq (rel IR, RA int)		
A_1	ν_1 in-phase terminal stretches	2229	(13)	[215]	2260-2271	m	[10.0]
	ν_2 sym central stretch	818	(0.5)	[5]	866-872	w	[0.6]
	ν_3 central bending	644	(2)	[1]	664-672	obscd	[1][a]
	ν_4 in-phase terminal bends	181	(0.3)	[6]	200-209	–[b]	[4]
A_2	ν_5 out-of-phase, out-of-plane bend	475	(0)	[1]	470-478	–	[0+]
B_1	ν_6 in-phase, out-of-plane bend	405	(6)	[0]	417-425	ms	[0]
B_2	ν_7 out-of-phase term stretches	2175	(105)	[42]	2203-2211	s	[1.9]
	ν_8 asym central stretch	1032	(138)	[2]	1055-1064	s	[n. obsd]
	ν_9 out-of-phase term bends	399	(1)	[0.5]	412-417	mw	[0+]

[a]Obscured in infrared and interference in Ra by anion bands. [b]Outside of frequency range of our spectrometer.

Figure Captions

Figure 1. An ORTEP diagram of $N_5^+Sb_2F_{11}^-$ showing the thermal ellipsoids at the 30% probability level.

Figure 2. An ORTEP diagram showing the side view of the eclipsed $Sb_2F_{11}^-$ anion. The thermal ellipsoids are drawn at the 30% probability level.

Figure 3. Space filling representation of $N_5^+Sb_2F_{11}^-$ showing the close packing of the N_5^+ cation within the $Sb_2F_{11}^-$ cavity.

Figure 4. A figure showing close range N···F contacts within the crystal lattice of $N_5^+Sb_2F_{11}^-$.

Figure 5. Infrared spectrum of solid $N_5^+SbF_6^-$ recorded as an AgBr pellet at room temperature. The band at 498 cm^{-1}, marked by an asterisk, is due to a small amount of $Sb_2F_{11}^-$.

Figure 6. Raman spectrum of solid $N_5^+SbF_6^-$ recorded at room temperature.

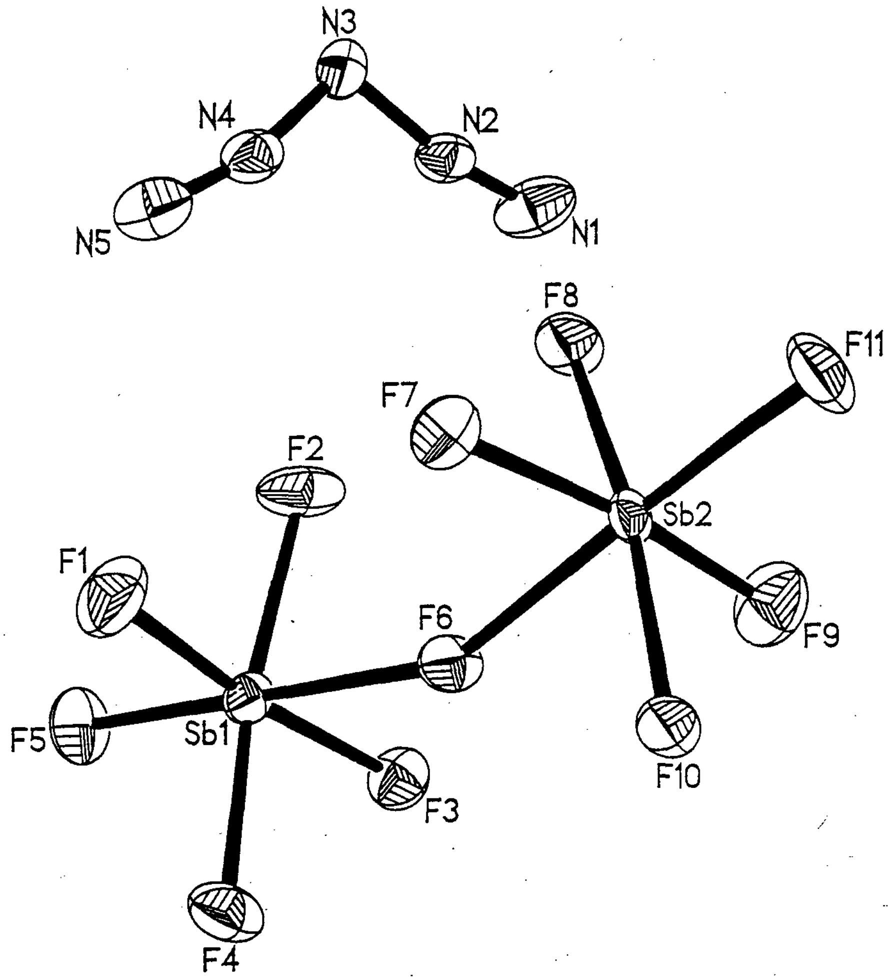


Figure 1

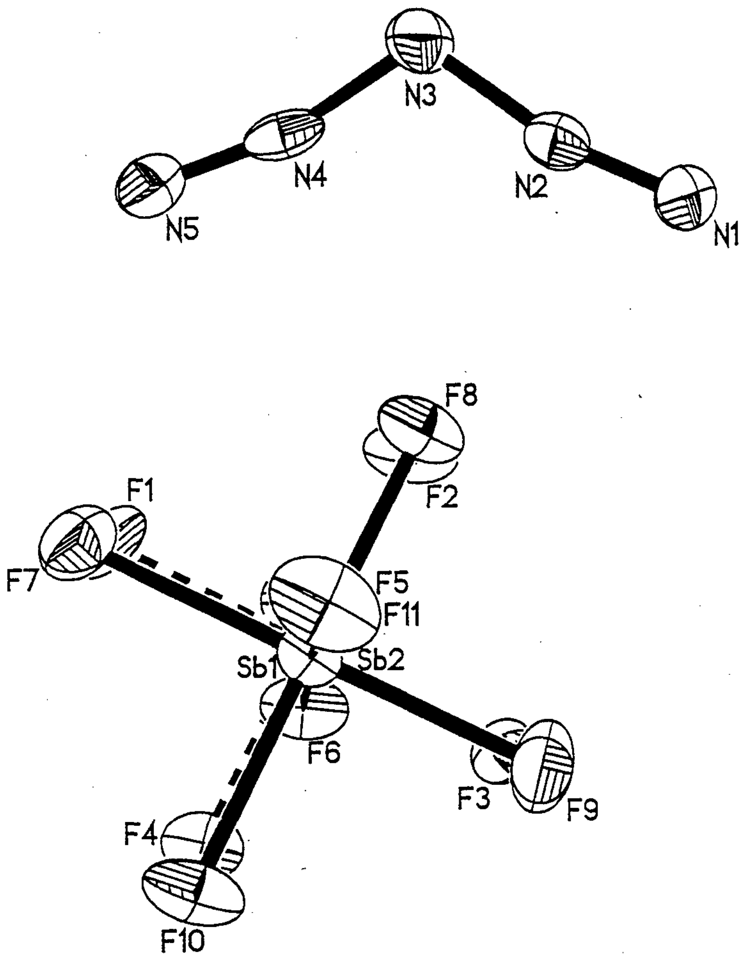


Figure 2

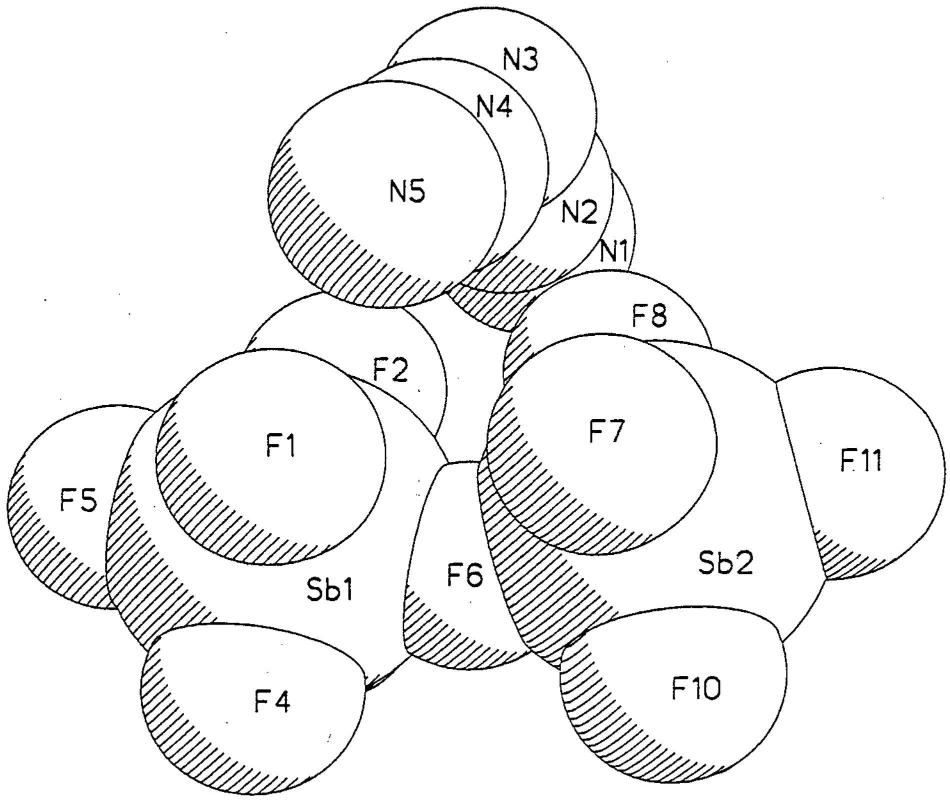


Figure 3

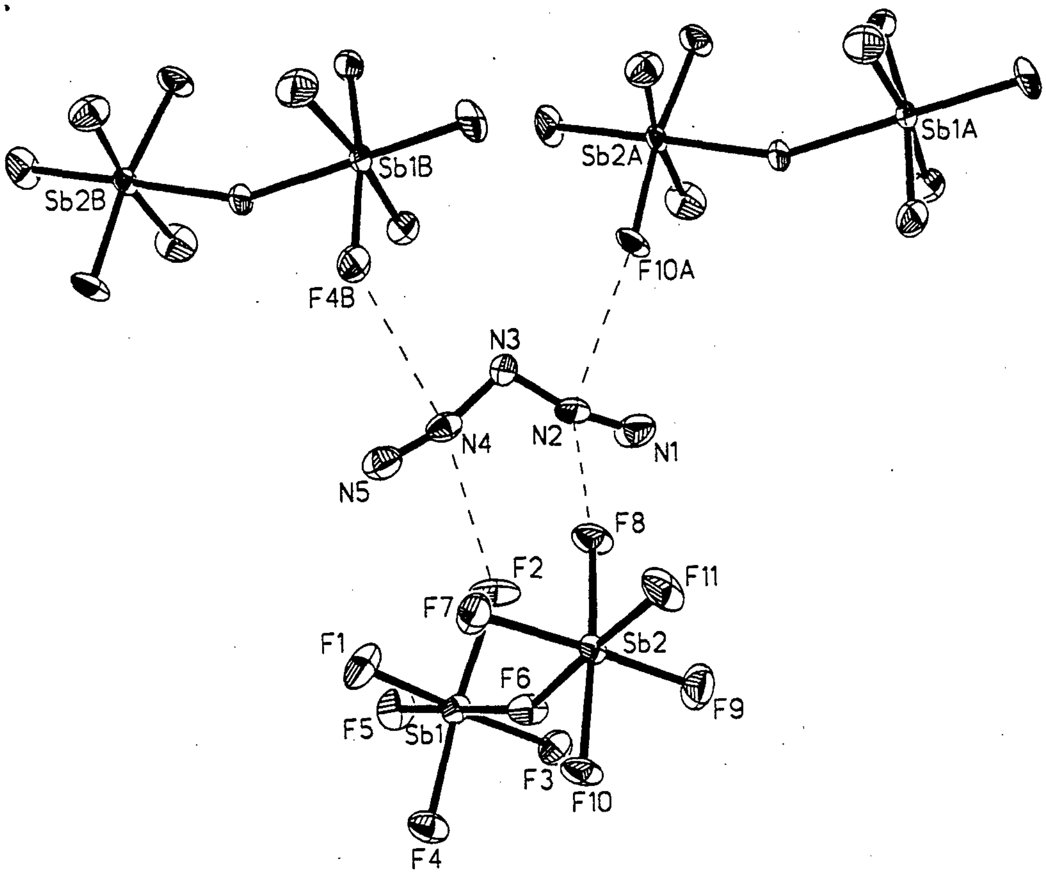
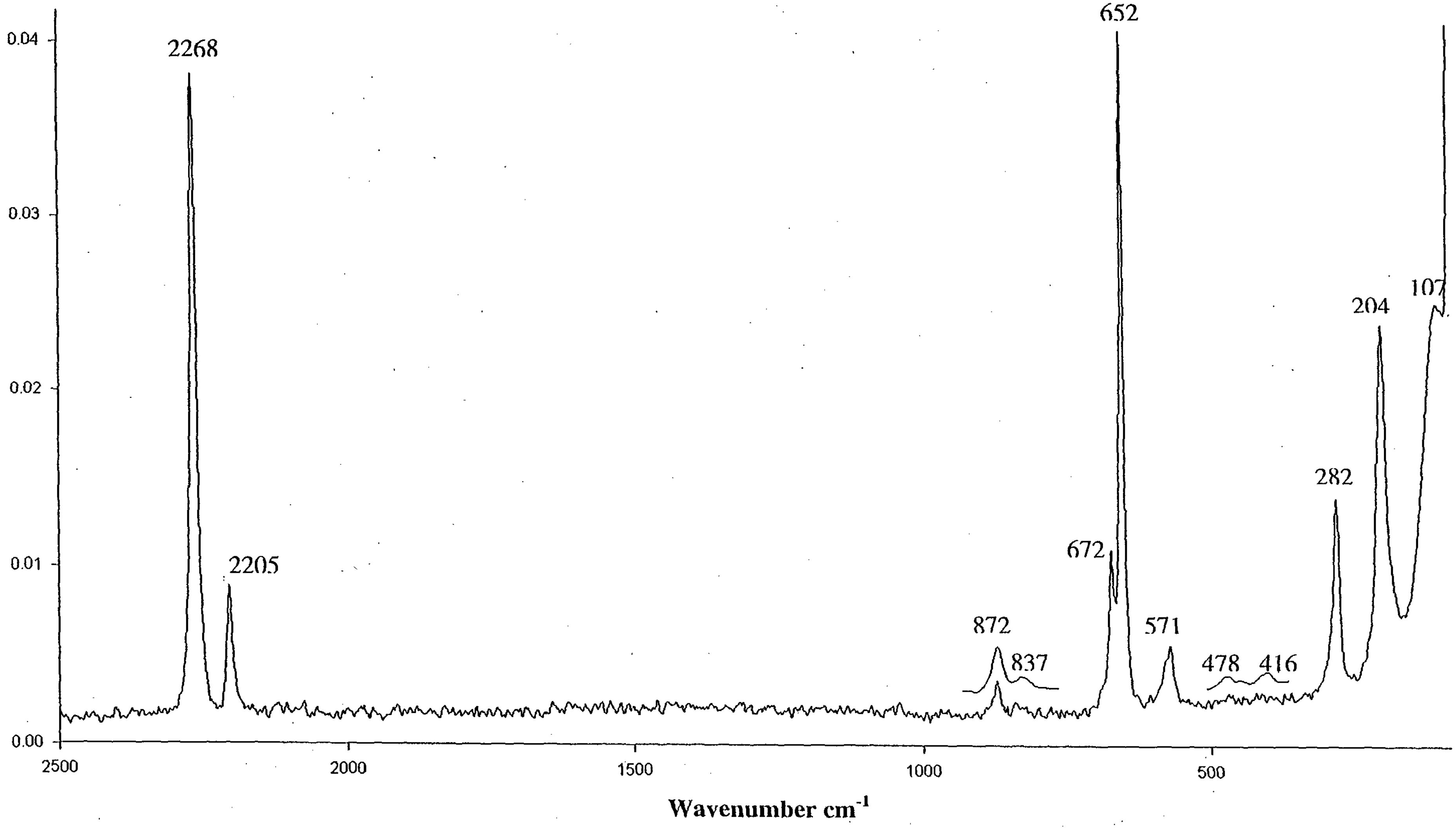

Sb2B
Sb1B
Sb2A
Sb1A
F4B
F10A
N3
N4
N2
N1
N5
F8
F2
F11
F7
Sb2
F1
F6
F9
F5
Sb1
F3
F10
F4

Figure 4

Raman Spectrum of $N_5^+SbF_6^-$



SUPPLEMENTARY MATERIAL

Polynitrogen Chemistry. Synthesis, Characterization, and Crystal Structure of Surprisingly Stable Fluoroantimonate Salts of N_5^+

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Abstract

The new N_5^+ salt, $N_5^+SbF_6^-$, was prepared from $N_2F^+SbF_6^-$ and HN_3 in anhydrous HF solution. The white solid is surprisingly stable, decomposing only at 70 °C, and is relatively insensitive to impact. Its vibrational spectrum exhibits all nine fundamentals with frequencies that are in excellent agreement with the theoretical calculations for a five-atomic V-shaped ion of C_{2v} symmetry. The $N_5^+Sb_2F_{11}^-$ salt was also prepared and its crystal structure was determined. The geometry previously predicted for free gaseous N_5^+ from theoretical calculations was confirmed within experimental error. The $Sb_2F_{11}^-$ anions exhibit an unusual geometry with eclipsed SbF_4 groups due to inter-ionic bridging with the N_5^+ cations. The N_5^+ cation is a powerful one-electron oxidizer. Its electron affinity falls between 11.0 and 12.08 eV because it readily oxidizes NO to NO^+ and NO_2 to NO_2^+ but fails to oxidize Xe or O_2.

Table 1. Crystal data and structure refinement for N5SB2F11.

Identification code	$N_5^+Sb_2F_{11}^-$	
Empirical formula	F11 N5 Sb2	
Formula weight	522.55	
Temperature	213(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 10.913(8) Å	α= 90°.
	b = 12.654(8) Å	β= 104.72(2)°.
	c = 16.675(11) Å	γ = 90°.
Volume	2227(3) $Å^3$	
Z	8	
Density (calculated)	3.117 Mg/m^3	
Absorption coefficient	4.995 mm^{-1}	
F(000)	1888	
Crystal size	0.26 x 0.10 x 0.05 mm^3	
Theta range for data collection	2.51 to 25.35°.	
Index ranges	-12<=h<=13, -15<=k<=15, -20<=l<=17	
Reflections collected	9125	
Independent reflections	2022 [R(int) = 0.0629]	
Completeness to theta = 25.35°	99.6 %	
Absorption correction	SADABS	
Max. and min. transmission	0.7883 and 0.3567	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2022 / 0 / 164	
Goodness-of-fit on F^2	1.122	
Final R indices [I>2sigma(I)]	R1 = 0.0678, wR2 = 0.1913	
R indices (all data)	R1 = 0.0785, wR2 = 0.2019	
Extinction coefficient	0.00026(18)	
Largest diff. peak and hole	4.329 and -2.102 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for N5SB2F11. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Sb(1)	2327(1)	5268(1)	5963(1)	32(1)
Sb(2)	1229(1)	2343(1)	6199(1)	28(1)
F(1)	930(11)	5546(9)	6373(7)	65(3)
F(2)	3187(10)	4839(8)	7017(5)	59(3)
F(3)	3652(9)	4765(7)	5561(6)	48(2)
F(4)	1368(9)	5453(7)	4889(5)	51(2)
F(5)	2906(11)	6638(7)	6101(6)	64(3)
F(6)	1691(8)	3755(6)	5813(5)	43(2)
F(7)	-17(9)	3062(8)	6548(6)	53(2)
F(8)	2340(9)	2672(7)	7217(5)	50(2)
F(9)	2592(10)	1837(9)	5844(6)	63(3)
F(10)	190(10)	2218(6)	5142(5)	49(2)
F(11)	854(10)	1003(6)	6539(6)	59(3)
N(1)	4750(14)	3132(16)	8389(9)	62(4)
N(2)	3965(12)	3518(11)	8581(7)	40(3)
N(3)	3138(14)	3881(10)	8954(8)	51(4)
N(4)	2229(13)	4376(10)	8441(7)	40(3)
N(5)	1369(14)	4828(13)	8136(8)	52(4)

Table 3. Bond lengths [Å] and angles [°] for N5SB2F11.

Sb(1)-F(5)	1.839(8)
Sb(1)-F(4)	1.845(8)
Sb(1)-F(2)	1.851(9)
Sb(1)-F(3)	1.854(9)
Sb(1)-F(1)	1.856(10)
Sb(1)-F(6)	2.031(7)
Sb(2)-F(10)	1.844(7)
Sb(2)-F(7)	1.849(9)
Sb(2)-F(9)	1.849(10)
Sb(2)-F(8)	1.866(8)
Sb(2)-F(11)	1.866(8)
Sb(2)-F(6)	2.007(7)
N(1)-N(2)	1.102(19)
N(2)-N(3)	1.303(19)
N(3)-N(4)	1.295(19)
N(4)-N(5)	1.107(19)
F(5)-Sb(1)-F(4)	95.4(4)
F(5)-Sb(1)-F(2)	94.8(4)
F(4)-Sb(1)-F(2)	169.9(4)
F(5)-Sb(1)-F(3)	95.4(5)
F(4)-Sb(1)-F(3)	89.7(4)
F(2)-Sb(1)-F(3)	89.3(5)
F(5)-Sb(1)-F(1)	93.6(5)
F(4)-Sb(1)-F(1)	91.0(5)
F(2)-Sb(1)-F(1)	88.4(5)
F(3)-Sb(1)-F(1)	170.8(4)
F(5)-Sb(1)-F(6)	179.9(5)
F(4)-Sb(1)-F(6)	84.7(4)
F(2)-Sb(1)-F(6)	85.1(4)
F(3)-Sb(1)-F(6)	84.5(4)
F(1)-Sb(1)-F(6)	86.4(4)
F(10)-Sb(2)-F(7)	91.2(5)
F(10)-Sb(2)-F(9)	90.8(5)

F(7)-Sb(2)-F(9)	170.7(5)
F(10)-Sb(2)-F(8)	171.3(4)
F(7)-Sb(2)-F(8)	87.9(4)
F(9)-Sb(2)-F(8)	88.8(5)
F(10)-Sb(2)-F(11)	94.8(4)
F(7)-Sb(2)-F(11)	96.7(5)
F(9)-Sb(2)-F(11)	92.2(5)
F(8)-Sb(2)-F(11)	93.9(4)
F(10)-Sb(2)-F(6)	85.0(3)
F(7)-Sb(2)-F(6)	85.9(4)
F(9)-Sb(2)-F(6)	85.3(5)
F(8)-Sb(2)-F(6)	86.3(4)
F(11)-Sb(2)-F(6)	177.5(4)
Sb(2)-F(6)-Sb(1)	155.0(4)
N(1)-N(2)-N(3)	168.1(15)
N(4)-N(3)-N(2)	111.2(11)
N(5)-N(4)-N(3)	166.3(14)

Symmetry transformations used to generate equivalent atoms.

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for N5SB2F11. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Sb(1)	46(1)	20(1)	33(1)	-4(1)	16(1)	-10(1)
Sb(2)	37(1)	15(1)	29(1)	0(1)	2(1)	-3(1)
F(1)	80(7)	58(6)	74(7)	-15(5)	48(6)	1(5)
F(2)	73(6)	69(7)	27(4)	2(4)	0(4)	-37(5)
F(3)	48(5)	50(6)	49(5)	-4(4)	19(4)	-5(4)
F(4)	64(6)	43(5)	41(5)	14(4)	5(4)	-8(4)
F(5)	101(8)	16(4)	73(7)	-10(4)	21(6)	-14(5)
F(6)	64(5)	29(4)	35(4)	1(3)	10(4)	-21(4)
F(7)	63(6)	48(6)	54(5)	0(4)	26(4)	10(5)
F(8)	63(6)	45(5)	30(4)	-1(4)	-12(4)	-6(4)
F(9)	52(6)	69(7)	70(6)	-14(5)	19(5)	15(5)
F(10)	78(6)	29(4)	25(4)	-2(3)	-16(4)	-7(4)
F(11)	80(7)	24(5)	61(6)	13(4)	-2(5)	-7(4)
N(1)	43(8)	103(14)	40(7)	-3(8)	8(6)	8(8)
N(2)	45(7)	46(7)	23(5)	0(5)	-1(5)	-6(6)
N(3)	77(10)	40(8)	39(7)	0(6)	19(7)	22(7)
N(4)	54(8)	37(7)	33(6)	-2(5)	18(6)	-12(6)
N(5)	51(8)	68(10)	41(7)	-1(7)	18(6)	-2(7)

Table 5. Torsion angles [°] for N5SB2F11.

F(10)-Sb(2)-F(6)-Sb(1)	156.0(13)
F(7)-Sb(2)-F(6)-Sb(1)	64.4(13)
F(9)-Sb(2)-F(6)-Sb(1)	-112.8(13)
F(8)-Sb(2)-F(6)-Sb(1)	-23.7(12)
F(11)-Sb(2)-F(6)-Sb(1)	-118(7)
F(5)-Sb(1)-F(6)-Sb(2)	47(100)
F(4)-Sb(1)-F(6)-Sb(2)	-154.6(13)
F(2)-Sb(1)-F(6)-Sb(2)	25.4(12)
F(3)-Sb(1)-F(6)-Sb(2)	115.2(13)
F(1)-Sb(1)-F(6)-Sb(2)	-63.3(13)
N(1)-N(2)-N(3)-N(4)	-173(8)
N(2)-N(3)-N(4)-N(5)	-179(100)

Symmetry transformations used to generate equivalent atoms.

Table 6 - Contact Distances(Angstrom) for N5SB2F11

Sb1	.N3_a	3.830(14)	F8	.F2	2.940(14)
Sb1	.F11_b	3.837(11)	F8	.N1	2.909(18)
Sb2	.F5_c	3.697(12)	F8	.N2	2.723(15)
F1	.N5	2.997(18)	F8	.N3	3.195(15)
F1	.N5_d	2.97(2)	F8	.N4	2.993(15)
F1	.F4_e	3.104(15)	F9	.N5_j	3.108(19)
F2	.F8	2.940(14)	F9	.F3_h	3.135(14)
F2	.N2	3.032(15)	F10	.N1_k	2.874(17)
F2	.N4	2.887(16)	F10	.N2_k	2.768(15)
F2	.N5	3.049(18)	F10	.N3_k	2.936(17)
F2	.F11_f	2.790(13)	F11	.Sb1_c	3.837(11)
F3	.N1_g	2.97(2)	F11	.F3_c	2.983(14)
F3	.N2_g	3.068(17)	F11	.F2_l	2.790(13)
F3	.N3_a	3.109(16)	F11	.N2_j	3.160(16)
F3	.F11_b	2.983(14)	F11	.N3_j	3.091(16)
F3	.F9_h	3.135(14)	F11	.N4_j	2.929(17)
F4	.F1_e	3.104(15)	N1	.F8	2.909(18)
F4	.F7_e	3.107(13)	N1	.N4	3.19(2)
F4	.N3_a	2.899(18)	N1	.F3_m	2.97(2)
F4	.N4_a	2.813(15)	N1	.N1_g	3.15(2)
F4	.N5_a	2.945(16)	N1	.F10_n	2.874(17)
F5	.Sb2_b	3.697(12)	N2	.F2	3.032(15)
F5	.F7_b	2.843(15)	N2	.F8	2.723(15)
F5	.N3_i	3.051(16)	N2	.N5	3.20(2)
F7	.N4_d	2.935(17)	N2	.F3_m	3.068(17)
F7	.N5_d	2.801(19)	N2	.F11_f	3.160(16)
F7	.F4_e	3.107(13)	N2	.F10_n	2.768(14)

F7	.F5_c	2.843(15)	N3	.F8	3.195(15)
N3	.Sb1_o	3.830(14)	N4	.F4_o	2.813(15)
N3	.F3_o	3.109(16)	N4	.F11_f	2.929(17)
N3	.F4_o	2.899(18)	N5	.F1	2.997(18)
N3	.F5_1	3.051(16)	N5	.F2	3.049(18)
N3	.F11_f	3.091(16)	N5	.N2	3.20(2)
N3	.F10_n	2.936(17)	N5	.F1_p	2.97(2)
N4	.F2	2.887(16)	N5	.F7_p	2.801(19)
N4	.F8	2.993(15)	N5	.N5_d	3.19(2)
N4	.N1	3.19(2)	N5	.F4_o	2.945(16)
N4	.F7_p	2.935(17)	N5	.F9_f	3.108(19)

Translation of Symmetry Code to Equiv.Pos

a =[4564.00] = x,1-y,-1/2+z
b =[5555.00] = 1/2+x,1/2+y,z
c =[5445.00] = -1/2+x,-1/2+y,z
d =[2556.00] = -x,y,3/2-z
e =[3566.00] = -x,1-y,1-z
f =[6556.00] = 1/2-x,1/2+y,3/2-z
g =[2656.00] = 1-x,y,3/2-z
h =[7556.00] = 1/2-x,1/2-y,1-z
i =[6556.00] = 1/2-x,1/2+y,3/2-z
j =[6546.00] = 1/2-x,-1/2+y,3/2-z
k =[8454.00] = -1/2+x,1/2-y,-1/2+z
l =[6546.00] = 1/2-x,-1/2+y,3/2-z
m =[2656.00] = 1-x,y,3/2-z
n =[8555.00] = 1/2+x,1/2-y,1/2+z
o =[4565.00] = x,1-y,1/2+z
p =[2556.00] = -x,y,3/2-z

Table 7. Observed and calculated structure factors for N5SB2F11

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
2	0	0	4431	4655	88	5	1	1	1750	2084	23	10	8	1	0	143	1	-5	3	2	2433	2482	56	-5	11	2	1110	1153	25
6	0	0	1529	1593	50	7	1	1	0	36	1	-9	9	1	0	11	1	-3	3	2	1420	1026	25	-3	11	2	1542	1533	19
8	0	0	2785	3166	23	9	1	1	1327	1399	38	-7	9	1	396	404	31	-1	3	2	4614	4116	58	-1	11	2	699	630	33
10	0	0	219	252	106	11	1	1	78	117	77	-5	9	1	905	788	31	1	3	2	3739	3562	56	1	11	2	466	405	31
12	0	0	500	514	72	-12	2	1	130	146	129	-3	9	1	2710	2456	74	3	3	2	686	771	12	3	11	2	1013	1008	21
1	1	0	173	5	27	-10	2	1	609	573	27	-1	9	1	1816	1282	18	5	3	2	523	578	16	5	11	2	906	981	22
3	1	0	672	774	10	-8	2	1	586	628	47	1	9	1	804	713	20	7	3	2	1158	1206	16	7	11	2	723	719	31
5	1	0	1086	1246	23	-6	2	1	675	646	16	3	9	1	1790	1765	23	9	3	2	555	548	22	-8	12	2	90	252	89
7	1	0	1082	1141	18	-4	2	1	832	650	37	5	9	1	2627	2614	53	11	3	2	0	130	1	-6	12	2	879	982	24
9	1	0	938	963	17	-2	2	1	1619	1334	14	7	9	1	1044	1071	31	-12	4	2	377	367	83	-4	12	2	617	545	27
11	1	0	740	749	33	0	2	1	403	302	9	9	9	1	188	179	92	-10	4	2	1606	1516	48	-2	12	2	548	496	24
0	2	0	272	310	27	2	2	1	0	20	1	-8	10	1	1514	1551	43	-8	4	2	536	487	21	0	12	2	512	493	28
2	2	0	4366	4048	44	4	2	1	443	412	19	-6	10	1	418	394	30	-6	4	2	1585	1274	24	2	12	2	0	21	1
4	2	0	2298	2369	19	6	2	1	1147	1280	22	-4	10	1	372	355	34	-4	4	2	62	52	61	4	12	2	774	785	24
6	2	0	1093	1189	25	8	2	1	0	101	1	-2	10	1	2332	2124	24	-2	4	2	2333	2118	25	6	12	2	887	942	28
8	2	0	834	859	16	10	2	1	368	383	30	0	10	1	1946	1651	40	0	4	2	849	709	11	-5	13	2	501	299	34
10	2	0	665	664	27	12	2	1	197	290	124	2	10	1	1155	1103	18	2	4	2	1224	1256	15	-3	13	2	686	293	24
12	2	0	785	756	49	-11	3	1	1013	1003	51	4	10	1	453	448	29	4	4	2	444	442	16	-1	13	2	1824	1848	32
1	3	0	2159	1900	29	-9	3	1	211	219	67	6	10	1	895	894	24	6	4	2	445	440	22	1	13	2	1267	1327	24
3	3	0	1331	1339	22	-7	3	1	2079	2086	26	8	10	1	431	447	40	8	4	2	1401	1394	26	3	13	2	706	762	26
5	3	0	84	81	84	-5	3	1	659	633	31	-7	11	1	676	645	31	10	4	2	1550	1580	30	5	13	2	609	630	32
7	3	0	798	841	16	-3	3	1	1991	1927	37	-5	11	1	569	436	32	-11	5	2	655	608	65	-4	14	2	116	165	115
9	3	0	1221	1226	24	-1	3	1	2221	1863	22	-3	11	1	1007	17	42	-9	5	2	0	94	1	-2	14	2	272	74	48
11	3	0	758	788	31	1	3	1	6532	5654	69	-1	11	1	1198	998	19	-7	5	2	934	817	17	0	14	2	349	339	40
0	4	0	12306	6174	419	3	3	1	1238	1325	13	1	11	1	1670	1682	19	-5	5	2	3175	2275	25	2	14	2	857	937	22
2	4	0	2362	2147	26	5	3	1	715	779	20	3	11	1	1639	1603	21	-3	5	2	3466	2770	50	4	14	2	339	418	43
4	4	0	403	344	19	7	3	1	957	1031	14	5	11	1	823	835	22	-1	5	2	636	513	14	-1	15	2	88	27	87
6	4	0	1136	1163	32	9	3	1	1810	1834	34	7	11	1	1146	1148	29	1	5	2	2171	1984	32	1	15	2	0	27	1
8	4	0	2386	2549	23	11	3	1	81	18	81	-6	12	1	1764	1872	20	3	5	2	2124	2062	24	-13	1	3	843	761	38
10	4	0	317	391	62	-12	4	1	670	632	35	-4	12	1	2159	2035	56	5	5	2	1413	1441	15	-11	1	3	0	114	1
12	4	0	537	563	39	-10	4	1	123	248	123	-2	12	1	122	107	122	7	5	2	192	242	47	-9	1	3	1005	886	22
1	5	0	1340	855	56	-8	4	1	738	780	18	0	12	1	282	268	42	9	5	2	209	252	45	-7	1	3	289	252	26
3	5	0	792	688	18	-6	4	1	1687	1705	32	2	12	1	1658	1717	22	11	5	2	404	410	43	-5	1	3	2756	2846	33
5	5	0	1110	1088	17	-4	4	1	2519	2433	20	4	12	1	1181	1246	22	-10	6	2	636	654	54	-3	1	3	185	179	53
7	5	0	1325	1344	28	-2	4	1	604	460	13	6	12	1	258	372	70	-8	6	2	60	37	60	-1	1	3	3052	3204	27
9	5	0	235	269	54	0	4	1	694	590	11	-5	13	1	222	218	74	-6	6	2	1322	1222	18	1	1	3	883	997	10
11	5	0	0	108	1	2	4	1	1600	1460	17	-3	13	1	1073	1180	20	-4	6	2	678	498	20	3	1	3	2182	2489	20
0	6	0	499	310	23	4	4	1	611	653	16	-1	13	1	1171	882	21	-2	6	2	4264	3970	42	5	1	3	77	90	77
4	6	0	3187	3129	26	6	4	1	486	477	19	1	13	1	465	383	57	0	6	2	492	399	19	7	1	3	2186	2344	29
6	6	0	292	306	28	8	4	1	843	843	18	3	13	1	1148	1192	27	2	6	2	1831	1735	17	9	1	3	0	72	1
8	6	0	519	529	26	10	4	1	291	325	42	5	13	1	1259	1347	30	4	6	2	764	768	20	11	1	3	461	476	27
10	6	0	455	423	48	12	4	1	0	100	1	-4	14	1	92	173	92	6	6	2	2466	2497	36	-12	2	3	230	207	98
1	7	0	1481	958	28	-11	5	1	791	762	62	-2	14	1	1606	1518	36	8	6	2	277	320	34	-10	2	3	0	80	1

h	k	l	10Fo	10Fc	10s
3	7	0	657	588	28
5	7	0	779	783	29
7	7	0	693	721	19
9	7	0	504	461	55
11	7	0	373	463	52
0	8	0	2675	2123	80
2	8	0	792	792	44
4	8	0	1597	1431	17
6	8	0	275	271	42
8	8	0	884	899	24
10	8	0	0	110	1
1	9	0	2173	1809	44
3	9	0	1099	1044	18
5	9	0	1017	950	26
7	9	0	644	570	38
9	9	0	693	684	30
0	10	0	1618	1342	26
2	10	0	668	482	30
4	10	0	1064	1032	20
6	10	0	287	277	42
8	10	0	492	529	35
1	11	0	1180	945	19
3	11	0	267	280	55
5	11	0	1211	1256	20
7	11	0	1164	1214	23
0	12	0	269	199	56
2	12	0	554	440	25
4	12	0	859	796	34
6	12	0	0	106	1
1	13	0	1450	1405	20
3	13	0	1174	1284	28
5	13	0	332	296	42
0	14	0	1480	1256	59
2	14	0	794	814	24
4	14	0	282	287	49
1	15	0	888	997	24
-11	1	1	751	749	41
-9	1	1	48	35	48
-7	1	1	1824	1820	37
-5	1	1	373	355	21
-3	1	1	3560	3426	41
-1	1	1	625	598	12
1	1	1	3475	3556	22
3	1	1	448	443	12

h	k	l	10Fo	10Fc	10s
-9	5	1	164	239	164
-7	5	1	1181	1171	17
-5	5	1	391	360	21
-3	5	1	3819	3311	48
-1	5	1	2014	1704	24
1	5	1	337	327	14
3	5	1	2255	2185	20
5	5	1	3034	3284	24
7	5	1	855	873	16
9	5	1	539	535	26
11	5	1	582	517	36
-10	6	1	1105	1095	61
-8	6	1	1245	1277	21
-6	6	1	704	644	18
-4	6	1	924	852	18
-2	6	1	5633	4261	82
0	6	1	1962	1805	18
2	6	1	1658	1564	35
4	6	1	1314	1335	16
6	6	1	928	968	18
8	6	1	269	250	39
10	6	1	860	860	30
-11	7	1	783	795	33
-9	7	1	232	180	76
-7	7	1	1240	1224	18
-5	7	1	1157	1046	18
-3	7	1	1345	1192	19
-1	7	1	2054	1764	21
1	7	1	5107	4753	37
3	7	1	1680	1596	19
5	7	1	217	187	38
7	7	1	1410	1431	21
9	7	1	1507	1506	26
11	7	1	222	253	96
-10	8	1	322	350	63
-8	8	1	282	332	52
-6	8	1	1987	1939	25
-4	8	1	3898	3500	59
-2	8	1	1251	992	17
0	8	1	24	9	23
2	8	1	2269	2138	18
4	8	1	1491	1467	19
6	8	1	556	570	20
8	8	1	712	708	23

h	k	l	10Fo	10Fc	10s
0	14	1	1540	1502	21
2	14	1	678	656	24
4	14	1	429	451	34
-1	15	1	645	414	28
1	15	1	584	615	28
-12	0	2	321	256	110
-10	0	2	1377	1394	36
-8	0	2	706	679	31
-4	0	2	572	571	24
0	0	2	1425	1732	28
2	0	2	5450	5801	71
4	0	2	132	159	131
6	0	2	875	968	25
8	0	2	1752	1738	30
10	0	2	1756	1817	43
12	0	2	0	142	1
-11	1	2	581	540	42
-9	1	2	728	671	19
-7	1	2	1689	1416	16
-5	1	2	2411	2248	38
-3	1	2	3744	3709	46
-1	1	2	3497	3359	55
1	1	2	2576	2743	12
3	1	2	1785	2078	25
5	1	2	854	967	13
7	1	2	294	281	29
9	1	2	217	241	50
11	1	2	121	210	120
-12	2	2	565	567	54
-10	2	2	786	759	25
-8	2	2	663	605	18
-6	2	2	2980	2727	39
-4	2	2	158	79	41
-2	2	2	4583	4626	91
0	2	2	1027	966	14
2	2	2	1778	1832	22
4	2	2	1094	1267	16
6	2	2	3410	3795	21
8	2	2	1282	1395	17
10	2	2	208	253	56
12	2	2	587	600	46
-11	3	2	43	175	42
-9	3	2	1402	1263	31
-7	3	2	2475	2172	22

h	k	l	10Fo	10Fc	10s
10	6	2	189	250	68
-11	7	2	0	170	1
-9	7	2	1088	985	36
-7	7	2	1214	1077	67
-5	7	2	371	314	25
-3	7	2	774	89	82
-1	7	2	3091	2934	34
1	7	2	2268	2088	35
3	7	2	288	253	23
5	7	2	709	675	17
7	7	2	733	720	20
9	7	2	593	623	25
-10	8	2	718	667	35
-8	8	2	158	210	157
-6	8	2	778	761	21
-4	8	2	312	274	33
-2	8	2	2212	2007	21
0	8	2	57	87	56
2	8	2	1544	1552	25
4	8	2	315	358	26
6	8	2	951	971	24
8	8	2	389	389	32
10	8	2	630	671	26
-9	9	2	256	330	79
-7	9	2	366	362	37
-5	9	2	1537	992	25
-3	9	2	2274	1607	39
-1	9	2	1552	1527	25
1	9	2	771	776	19
3	9	2	1622	1499	22
5	9	2	999	1009	20
7	9	2	720	718	23
9	9	2	0	80	1
-8	10	2	368	393	41
-6	10	2	616	547	25
-4	10	2	262	169	48
-2	10	2	1216	1119	24
0	10	2	343	320	33
2	10	2	1074	1054	28
4	10	2	80	149	80
6	10	2	760	708	29
8	10	2	268	349	65
-9	11	2	512	559	42
-7	11	2	728	646	77

h	k	l	10Fo	10Fc	10s
-8	2	3	331	321	26
-6	2	3	1580	1457	21
-4	2	3	1215	1299	19
-2	2	3	894	915	19
0	2	3	1601	1571	30
2	2	3	1524	1554	22
4	2	3	710	782	15
6	2	3	783	823	14
8	2	3	774	822	18
10	2	3	0	161	1
-11	3	3	263	297	65
-9	3	3	1921	1799	48
-7	3	3	943	984	20
-5	3	3	2109	1671	33
-3	3	3	1386	1424	24
-1	3	3	4552	4440	31
1	3	3	68	50	68
3	3	3	1602	1746	21
5	3	3	263	286	24
7	3	3	1513	1577	25
9	3	3	731	720	19
11	3	3	943	967	21
-12	4	3	300	242	81
-10	4	3	216	121	81
-8	4	3	848	609	21
-6	4	3	615	462	20
-4	4	3	1990	1768	39
-2	4	3	483	440	16
0	4	3	89	57	89
2	4	3	730	677	14
4	4	3	1237	1303	14
6	4	3	1537	1689	21
8	4	3	0	96	1
10	4	3	0	25	1
-11	5	3	660	668	67
-9	5	3	282	233	62
-7	5	3	1193	1184	18
-5	5	3	3704	3610	39
-3	5	3	1484	1378	23
-1	5	3	2061	1854	48
1	5	3	143	119	36
3	5	3	1428	1392	13
5	5	3	693	721	17
7	5	3	1924	2041	20

h	k	l	10Fo	10Fc	10s
9	5	3	267	297	36
11	5	3	93	224	93
-12	6	3	181	198	180
-10	6	3	0	163	1
-6	6	3	1821	1535	17
-4	6	3	474	394	28
-2	6	3	1776	1671	20
0	6	3	4881	4330	16
2	6	3	3709	3458	43
4	6	3	1151	1135	13
6	6	3	498	525	19
8	6	3	1323	1333	33
10	6	3	511	557	27
-11	7	3	511	587	42
-9	7	3	1701	1732	61
-7	7	3	1399	1271	39
-5	7	3	334	258	27
-3	7	3	1040	977	22
-1	7	3	3481	3298	37
1	7	3	192	185	27
3	7	3	2269	2132	24
5	7	3	464	438	18
7	7	3	771	817	18
9	7	3	754	787	23
-10	8	3	692	623	59
-8	8	3	108	203	108
-6	8	3	259	186	45
-4	8	3	2839	2516	30
-2	8	3	3512	3491	50
0	8	3	885	773	17
2	8	3	1054	932	15
4	8	3	2908	2901	23
6	8	3	1891	1907	23
8	8	3	214	211	60
10	8	3	0	45	1
-9	9	3	246	287	84
-7	9	3	1040	1103	21
-5	9	3	1916	1881	39
-3	9	3	602	547	26
-1	9	3	661	548	19
1	9	3	375	338	21
3	9	3	956	914	18
5	9	3	551	508	23
7	9	3	1050	1111	20
9	9	3	445	502	33

h	k	l	10Fo	10Fc	10s
-4	0	4	1171	1234	30
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2	0	4	1393	1520	22
4	0	4	2163	2450	48
6	0	4	786	910	25
8	0	4	1343	1484	22
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-1	1	4	2734	2933	36
1	1	4	2333	2497	22
3	1	4	1060	1160	15
5	1	4	1942	2082	17
7	1	4	1554	1690	17
9	1	4	929	956	19
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-8	2	4	81	90	81
-6	2	4	3205	3281	73
-4	2	4	5768	6222	56
-2	2	4	2173	2426	42
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6	2	4	204	169	33
8	2	4	1371	1488	24
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-8	8	4	966	885	36
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6	8	4	286	284	32
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-7	9	4	621	258	42
-5	9	4	1082	1063	24
-3	9	4	890	890	21
-1	9	4	937	832	21
1	9	4	1140	1143	17
3	9	4	1259	1264	15
5	9	4	949	963	19
7	9	4	379	422	45
9	9	4	426	455	34
-8	10	4	748	621	32
-6	10	4	514	401	29
-4	10	4	829	796	20
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6	10	4	0	39	1
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-9	11	4	391	259	49
-7	11	4	1161	1133	26
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-3	11	4	1498	1486	22
-1	11	4	1170	1075	21
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7	11	4	685	751	54
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-12	4	5	719	638	42
-10	4	5	204	166	57
-8	4	5	1022	978	28
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-4	4	5	599	638	16
-2	4	5	2341	2260	28
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6	4	5	1561	1614	17
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-7	5	5	805	710	31
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9	5	5	703	668	21
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-4	6	5	782	763	18
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2	12	5	846	838	25
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-3	13	5	1472	1554	36
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3	13	5	917	966	27
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-4	14	5	392	393	35
-2	14	5	980	1106	22
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-8	0	6	235	261	68
-6	0	6	3517	4033	85
-4	0	6	1484	1629	19
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-5	1	6	2237	2525	30
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-9	11	3	1258	1322	59
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-3	11	3	844	889	24
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5	11	3	201	260	81
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-8	12	3	135	237	134
-6	12	3	0	69	1
-4	12	3	2134	2081	29
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4	12	3	1985	2006	20
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-2	14	3	0	92	1
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-12	0	4	102	107	101
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-2	4	4	1731	1664	19
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-1	5	4	597	587	14
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-10	6	4	616	393	40
-8	6	4	0	122	1
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-9	7	4	311	101	53
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-5	7	4	1072	982	20
-3	7	4	1354	1210	20
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-9	1	5	0	7	1
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4	2	5	670	730	14
6	2	5	76	131	76
8	2	5	457	443	23
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-8	8	5	253	284	57
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6	8	5	1930	1977	32
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3	9	5	1902	1844	22
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h	k	l	10Fo	10Fc	10s
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7	1	6	530	541	23
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2	2	6	444	455	18
4	2	6	2973	3149	59
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8	2	6	418	460	25
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-3	3	6	23	79	23
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7	3	6	220	252	36
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7	5	6	767	788	16
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-4	6	6	920	925	23
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-5	7	6	293	269	40
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-4	8	6	144	144	117
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-9	9	6	320	379	51
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9	1	7	242	272	43
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-10	2	7	114	240	114
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-6	2	7	892	836	19
-4	2	7	658	638	17
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-4	4	7	1877	1969	39
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h	k	l	10Fo	10Fc	10s
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2	10	7	1595	1670	29
4	10	7	655	590	29
6	10	7	0	32	1
-7	11	7	78	127	78
-5	11	7	730	654	28
-3	11	7	54	93	54
-1	11	7	276	292	50
1	11	7	992	1013	56
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-6	12	7	262	340	69
-4	12	7	2171	2248	25
-2	12	7	1740	1832	32
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-5	13	7	150	208	149
-3	13	7	0	87	1
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1	13	7	411	408	52
3	13	7	0	98	1
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-12	0	8	229	223	82
-10	0	8	929	880	32
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-6	0	8	1293	1402	30
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-2	0	8	614	694	27
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4	0	8	2846	2901	55
6	0	8	72	174	72
8	0	8	594	580	58
10	0	8	930	870	29
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-11	5	8	471	428	46
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-5	5	8	580	523	21
-3	5	8	1850	1951	20
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3	5	8	1237	1266	13
5	5	8	1543	1602	16
7	5	8	623	608	18
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-8	6	8	709	688	24
-6	6	8	578	524	24
-4	6	8	1802	1814	33
-2	6	8	139	132	47
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6	6	8	204	256	41
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-7	7	8	1106	1143	19
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-1	7	8	43	139	43
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5	7	8	467	431	23
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6	2	9	217	211	39
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-9	3	9	787	781	22
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3	3	9	703	696	26
5	3	9	1168	1172	14
7	3	9	232	273	42
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9	5	9	804	805	23

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
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3	11	9	303	264	74
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-2	12	9	1527	1713	139
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-3	13	9	1073	1144	30
-1	13	9	771	749	41
1	13	9	272	262	77
-12	0	10	0	161	1
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-8	0	10	763	796	27
-6	0	10	2618	2936	62
-4	0	10	1703	1941	20
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-7	7	10	229	269	70
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4	4	11	264	231	33
6	4	11	161	211	80
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-4	6	11	925	899	16
-2	6	11	6	83	6
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-3	7	11	897	936	22
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5	7	11	315	315	31
7	7	11	123	194	122
-10	8	11	866	907	27
-8	8	11	256	264	59
-6	8	11	718	739	33
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6	8	11	350	354	33

h	k	l	10Fo	10Fc	10s
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6	2	12	1503	1447	44
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-9	3	12	659	594	26
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-7	5	12	192	195	62
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3	5	12	386	377	24
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7	5	12	127	157	127
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-6	4	13	0	66	1
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-2	4	13	1551	1628	25
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-1	5	13	947	966	66
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h	k	l	10Fo	10Fc	10s
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7	3	10	132	166	132
9	3	10	434	408	33
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-11	5	10	810	782	26
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7	5	10	802	780	24
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-6	6	10	186	173	73
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h	k	l	10Fo	10Fc	10s
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-4	12	10	441	447	52
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-1	13	10	646	692	37
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-9	1	11	1071	1086	18
-7	1	11	261	243	48
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3	1	11	941	930	18
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-8	2	11	690	727	23
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4	2	11	107	49	106
6	2	11	286	254	40
8	2	11	0	62	1
-11	3	11	192	202	87
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-3	3	11	92	98	91
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-8	4	11	41	195	41
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-7	9	11	298	316	66
-5	9	11	101	81	101
-3	9	11	1305	1355	49
-1	9	11	2208	2272	35
1	9	11	789	766	28
3	9	11	440	419	34
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-8	10	11	1434	1556	31
-6	10	11	1256	1303	30
-4	10	11	217	208	87
-2	10	11	69	48	69
0	10	11	1261	1301	25
2	10	11	449	404	39
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-7	11	11	512	521	48
-5	11	11	921	970	35
-3	11	11	494	518	70
-1	11	11	287	351	53
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3	11	11	1319	1290	47
-4	12	11	1329	1472	45
-2	12	11	796	782	39
0	12	11	772	795	47
-12	0	12	0	126	1
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-8	0	12	966	1035	31
-6	0	12	0	54	1
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-2	0	12	356	353	29
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2	0	12	1032	1022	31
4	0	12	2589	2415	36
6	0	12	243	267	62
8	0	12	244	270	101
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-7	1	12	1028	1056	26
-5	1	12	2266	2414	49
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0	6	12	1443	1452	26
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6	6	12	775	726	22
-9	7	12	0	43	1
-7	7	12	996	1120	27
-5	7	12	941	969	35
-3	7	12	187	192	49
-1	7	12	297	377	56
1	7	12	1209	1188	23
3	7	12	524	510	22
5	7	12	418	412	27
-8	8	12	727	709	34
-6	8	12	288	279	67
-4	8	12	747	722	29
-2	8	12	390	393	27
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2	8	12	343	372	31
4	8	12	643	624	21
-7	9	12	562	554	42
-5	9	12	343	347	79
-3	9	12	768	817	37
-1	9	12	222	222	62
1	9	12	878	907	24
3	9	12	349	373	39
-6	10	12	0	84	1
-4	10	12	573	564	37
-2	10	12	0	105	1
0	10	12	590	568	29
2	10	12	437	421	33
-5	11	12	167	199	167
-3	11	12	1257	1321	42
-1	11	12	449	490	38
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-11	1	13	859	886	27
-9	1	13	213	174	52
-7	1	13	783	836	25
-5	1	13	802	846	16
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2	6	13	1712	1632	23
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-7	7	13	1475	1593	28
-5	7	13	538	517	57
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-8	8	13	796	846	37
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-7	9	13	0	164	1
-5	9	13	772	846	39
-3	9	13	941	996	33
-1	9	13	274	311	55
1	9	13	563	571	37
3	9	13	224	291	64
-6	10	13	1323	1443	32
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-2	10	13	151	125	151
0	10	13	777	784	26
2	10	13	1762	1659	24
-5	11	13	340	308	71
-3	11	13	0	221	1
-1	11	13	251	340	62
-10	0	14	245	257	77
-8	0	14	949	1002	25
-6	0	14	2205	2313	80
-4	0	14	950	973	22
-2	0	14	182	229	56
0	0	14	963	825	48
2	0	14	1281	1325	34
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h	k	l	10Fo	10Fc	10s
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-9	1	14	0	37	1
-7	1	14	285	313	37
-5	1	14	617	597	20
-3	1	14	1028	1006	21
-1	1	14	840	826	40
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-4	2	14	2133	2161	38
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-9	3	14	0	98	1
-7	3	14	159	223	88
-5	3	14	801	822	27
-3	3	14	709	711	29
-1	3	14	257	251	48
1	3	14	705	618	33
3	3	14	1299	1286	19
5	3	14	1193	1157	22
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-8	4	14	1132	1135	23
-6	4	14	1730	1895	54
-4	4	14	212	259	53
-2	4	14	489	480	32
0	4	14	1174	1057	38
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6	4	14	702	708	24
-9	5	14	340	357	51
-7	5	14	632	602	34
-5	5	14	24	121	24
-3	5	14	92	72	91
-1	5	14	983	1038	46
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h	k	l	10Fo	10Fc	10s
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-8	8	14	478	480	71
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-2	8	14	280	331	54
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-7	9	14	301	386	103
-5	9	14	522	624	45
-3	9	14	120	138	120
-1	9	14	871	865	31
1	9	14	420	439	37
-6	10	14	563	541	45
-4	10	14	173	81	156
-2	10	14	579	655	39
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-7	1	15	267	276	56
-5	1	15	1096	1094	17
-3	1	15	968	937	29
-1	1	15	1142	1167	33
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4	6	15	957	934	21
-7	7	15	1097	1118	29
-5	7	15	1716	1735	29
-3	7	15	777	812	23
-1	7	15	340	315	64
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3	7	15	1201	1160	19
-6	8	15	97	96	96
-4	8	15	783	727	39
-2	8	15	0	76	1
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-5	9	15	164	265	163
-3	9	15	1066	1108	29
-1	9	15	1619	1534	57
1	9	15	411	399	44
-2	10	15	193	249	113
-10	0	16	208	163	93
-8	0	16	198	221	89
-6	0	16	1135	1065	22
-4	0	16	2356	2291	69
-2	0	16	796	735	52
0	0	16	292	321	71
2	0	16	1588	1500	42
4	0	16	2015	1944	66
-9	1	16	1117	1075	18
-7	1	16	1073	1029	16
-5	1	16	1047	1030	28
-3	1	16	787	732	35
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h	k	l	10Fo	10Fc	10s
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-1	5	16	757	676	24
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-8	6	16	783	818	31
-6	6	16	125	27	125
-4	6	16	153	201	152
-2	6	16	775	756	41
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-3	9	16	649	618	38
-1	9	16	0	122	1
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-7	1	17	908	882	41
-5	1	17	500	468	44
-3	1	17	992	891	35
-1	1	17	634	492	42
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-6	2	17	649	615	28
-4	2	17	192	141	192
-2	2	17	73	66	73
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-9	3	17	329	306	53
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-5	3	17	519	496	30
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h	k	l	10Fo	10Fc	10s
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-2	6	17	298	321	46
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-8	0	18	966	866	50
-6	0	18	1105	966	50
-4	0	18	135	14	135
-2	0	18	744	597	59
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-7	1	18	702	613	39
-5	1	18	722	603	39
-3	1	18	1067	983	36
-1	1	18	1318	1107	37
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-5	3	18	681	617	38
-3	3	18	929	773	36
-1	3	18	805	691	34
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-4	6	18	428	419	37
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-8	6	14	267	340	76	-2	4	15	0	37	1	-9	3	16	717	703	30	3	3	17	1101	1008	23	-4	2	19	428	299	79
-6	6	14	122	128	122	0	4	15	815	822	34	-7	3	16	989	980	23	-8	4	17	833	827	30	-2	2	19	307	199	86
-4	6	14	1089	1122	29	2	4	15	262	303	35	-5	3	16	599	593	28	-6	4	17	121	201	121	0	2	19	656	5	31
-2	6	14	1691	1740	28	4	4	15	0	88	1	-3	3	16	211	247	71	-4	4	17	668	597	34	-5	3	19	867	864	36
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